Toxicity Characteristic Leaching Procedure (TCLP) for VOCs, SVOCs, Chlorinated Pesticides and Herbicides, and Metals by SW-846 Method 1311 and Analysis

Table 1A. Summary of Holding Times and Preservation for TCLP Volatile Organic Compounds (VOCs) by SW-846 Method 1311

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation** [b]
Volatile Organic Compounds (VOCs) in Water	Technical for TCLP Extraction: 14 days from collection; Contract for TCLP Extraction: 10 days from receipt at laboratory; Technical and Contract Analysis: 14 days from date of TCLP extraction to analysis.	Cool to 4°C ±2°C
VOCs in Soil [c],[d]	Technical and Contract for TCLP Extraction: 48 hours cumulative from collection; (7 days if frozen) [c] Technical and Contract of TCLP Extract: 7 days from date of TCLP extraction to analysis.	Cool to 4°C ±2°C; sealed zero headspace containers. [c]

[a] Individual target compounds are listed in Table 1B.
[b] Preservatives should not be added to samples before extraction.
[c] Freezing of soil samples requires contract approval.
[d] Freezing of En Core ™ samplers requires contract approval.

Data Calculations and Reporting Units:

Determine the percent solid as specified in Section 7.1 of SW-846 Method 1311 and report the result as percent solid. Extract the second sample/aliquot according to Section 7.3 of SW-846 Method 1311.

Calculate the response factor (RF) and the concentration of individual analytes according to the equations specified in Sections 7.3 and 7.5 of SW-846 Method 8260B, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), (Revision 2). Report final analyte concentration in units of micrograms per liter (µg/L). Report results that are less than 10 µg/L to 1 significant figure, and results that are greater than or equal to 10 µg/L to 2 significant figures.

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 1B. Target Compound List, Contract Required Quantitation Limits

(CRQLs), and Regulatory and Spiking Levels for TCLP VOCs by GC/MS Method 8260B

Analyte	**CRQL µg/L**	**Regulatory Level mg/L**	**Spiking Level µg/L**
Benzene	10	0.5	50
Carbon tetrachloride	10	0.5	50
Chlorobenzene	10	100	50
Chloroform	10	6.0	50
1,4-Dichlorobenzene	10	7.5	50
1,2-Dichloroethane	10	0.5	50
1,1-Dichloroethene	10	0.7	50
Methyl ethyl ketone	10	200	50
Tetrachloroethene	10	0.7	50
Trichloroethene	10	0.5	50
Vinyl chloride	10	0.2	50

Table 2. Summary of Calibration Procedures for TCLP VOCs by SW-846 Method 8260B

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with 4-bromofluorobenzene (BFB)	Beginning of each 12 hour period during which standards and samples are analyzed	Ion abundance criteria in Table 4 of SW-846 Method 8260B	1. Identify the problem. 2. MS tune criteria must be met before calibration
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) [a, b, c]	Initially; whenever required, due to failure of CCV	RSD for RFs #20%;	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Continuing Calibration Verification (CCV)	Following ICV, every 12-hour, and end of run	%D between RF of CCV and avg RFs from ICAL #±15%	1. Recalibrate and verify 2. Reanalyze samples back to last good CCV
System Performance Check Compound (SPCC)	With ICAL or CCV	RF for chloromethane, 1,1-dichloroethane, bromoform, $0.10; chlorobenzene, 1,1,2,2-tetrachloroethane, $0.30	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Calibration Check Compounds (CCC)	With ICAL or CCV	RSD for RFs #30%	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Internal Standards	Every standard, sample, blank, and QC sample	IS area within a factor of two of the IS area in the associated CCV (-50% to +100%)	1. Investigate the system; 2. Re-analyze all samples analyzed during a system malfunction
Retention time evaluation of CCV standards	Each analysis of CCV standard	±3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $5:1. If this requirement cannot be met, the laboratory must submit a method detection limit (MDL) study as part of the data package.
[b] ICAL and CCV standards must contain all target analytes listed in Table 1B.
[c] Report the retention time (RT) window for each analyte. Determine RT windows as ±3 x the standard deviation (SD) of the average initial calibration RT for each analyte.

Table 3. Summary of Internal Quality Control Procedures for TCLP VOCs BY SW-846 Method 8260B

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Each 12-hour time period, minimum of one per SDG [a] or one per batch of extraction fluid	< CRQL for each compound	1. Investigate the source of contamination and document. 2. Reextract and/or reanalyze all samples processed with a blank that is out of control.
Matrix Spike and Matrix Spike Duplicate (MS/MSD) [b]	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	Sample extract: 75-125% of expected value; #25% RPD between MS and MSD	1. Report in case narrative
Surrogate Spikes [b, c]	Every sample, QC sample, standard and method blank	Sample extract: 85-115% of expected value, except for 1,2-dichloroethane (75-115%)	1. Reanalyze all samples with non-compliant surrogate recoveries
Laboratory Control Sample (LCS)	One per SDG	Sample extract: 70-130% of expected value	1. Investigate the source of problem and document. 2. Reanalyze all samples processed with a LCS that is out of control.

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.
[b] Spiking solution to be added after the TCLP extraction, immediately preceding analysis.
[c] Toluene-d_8, BFB, 1,2-dichloroethane-d_4, and dibromofluoromethane

Dilute and reanalyze samples which contain one or more target analytes at concentrations above the initial calibration range. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Table 4A. Summary of Holding Times and Preservation for TCLP SVOCs by SW-846 Method 1311

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation** [b]
Semivolatile Organic Compounds (SVOCs)	Technical for TCLP Extraction: 14 days from collection; Contract for TCLP Extraction: 10 days from receipt at laboratory; Technical and Contract of TCLP Extract: 7 days from date of TCLP extraction to preparative extraction; Technical for Analysis: 40 days from preparative extraction; Contract for Analysis: 35 days from preparative extraction.	Cool to 4°C ±2°C

[a] Individual target compounds are listed in Table 4B.
[b] Preservatives should not be added to samples before extraction.

Data Calculations and Reporting Units:

Determine the percent solid as specified in Section 7.1 of SW-846 Method 1311 and report the result as percent solid. Extract the second sample/aliquot according to Section 7.3 of SW-846 Method 1311.

Analyze the extract for SVOCs by SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), (Revision 3.0). Calculate the concentration of individual analytes according to Section 7.7.2 of EPA Method 8270C. Report final analyte concentration in units of micrograms per liter (µg/L). Report results that are less than 10 µg/L to 1 significant figure, and results that are greater than or equal to 10 µg/L to 2 significant figures.

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 4B. ***Target Compound List, Contract Required Quantitation Limits (CRQLs), and Regulatory and Spiking Levels for TCLP SVOCs by SW-846 Method 8270C***

Analyte	CRQL µg/L	Regulatory Level mg/L	Spiking Level µg/L
o-Cresol (2-Methylphenol)	10	200	50
m-Cresol (3-Methylphenol)	10	200	50
p-Cresol (4-Methylphenol)	10	200	50
2,4-Dinitrotoluene	10	0.13	50
Hexachlorobenzene	10	0.13	50
Hexachloro-1,3-butadiene	10	0.5	50
Hexachloroethane	10	3.0	50
Nitrobenzene	10	2.0	50
Pentachlorophenol	25	100	75
Pyridine	25	5.0	75
2,4,5-Trichlorophenol	25	400	75
2,4,6-Trichlorophenol	10	2.0	50

Table 5. **Summary of Calibration Procedures for TCLP SVOCs by SW-846 Method**

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with decafluorotriphenyl-phosphine (DFTPP)	Beginning of each 12-hour period during which standards and samples are analyzed	Ion abundance criteria in Table 3 of Method 8270C, Revision 3.0	1. Identify the problem. 2. MS tune criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) [a, b, c]	Initially; whenever required, due to failure of CCV	%RSD for RRFs #30%; or correlation coefficient (r) generated by the linear regression must be $0.99 for all analytes	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Continuing Calibration Verification (CCV) [d]	Beginning of every 12-hour period, and end of run	%D between RRF of CCV and avg RRFs from ICAL #30%; or ±30% of true value for linear regressiion	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Integrated areas of Internal Standards (IS)	Each analysis	Area must be within -50 to 100 percent. Retention time ± 0.33 minutes of CCV IS retention times.	1. Re-analyze samples with internal standard -50 percent and greater than 100 percent
Retention time evaluation of all standard, surrogate, and sample analytes	Each analysis	±3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples out of control limits

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $5:1. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

[b] ICAL and continuing CAL standards must contain all target analytes listed in Table 4B.

[c] Report the retention time window for each analyte. Determine retention time windows as ±3 x the standard deviation of the average initial calibration retention time for each analyte.

[d] If some compounds are beyond the control limits of the CCV and these target compounds are detected in samples and 10 percent or less of these analytes are beyond control limits, a single point calibration may be used to quantify the out-of-control analytes.

Table 6. Summary of Internal Quality Control Procedures for TCLP SVOCs by SW-846 Method 8270C

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Each 12-hour time period, minimum of one per SDG [a] or one per batch of extraction fluid	< CRQL for each compound	1. Investigate the source of contamination and document. 2. Re-analyze all samples processed with a blank that is out of control.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	MS and MSD recoveries (65-135%) and RPD 30%	1. Report in case narrative
Surrogate Spikes [b]	Every sample, standard and method blank	Surrogate recoveries within laboratory limits	1. Re-analyze all samples with non-compliant surrogate recoveries
Laboratory Control Sample (LCS) [b]	One per SDG	LCS recoveries within laboratory limits	1. Investigate the source of problem and document. 2. Re-analyze all samples processed with a LCS that is out of control.

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

[b] Within 3 sigma of laboratory control charts. The laboratory should submit the control charts.

Dilute and re-analyze samples which contain one or more target analytes at concentrations above the initial calibration range. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Table 7A. Summary of Holding Times and Preservation for TCLP Chlorinated Pesticides by SW-846 Method 1311

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation** [b]
Chlorinated Pesticides	Technical for TCLP Extraction: 14 days from collection; Contract for TCLP Extraction: 10 days from receipt at laboratory; Technical and Contract of TCLP Extract: 7 days from date of TCLP extraction to preparative extraction; Technical for Analysis: 40 days from preparative extraction; Contract for Analysis: 35 days from preparative extraction.	Cool to 4°C ±2°C

[a] Individual target compounds are listed in Table 7B.
[b] Preservatives should not be added to samples before extraction.

Data Calculations and Reporting Units:

Determine the percent solid as specified in Section 7.1 of SW-846 Method 1311 and report the result as percent solid. Extract the second sample/aliquot according to Section 7.3 of SW-846 Method 1311.

Calculate the calibration factors (CF) of single component pesticides according to Section 7.4 of SW-846 Method 8081, Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique, (Revision 0). Calculate sample results using the analyte CFs from the midpoint standard of the associated initial calibration curve. Perform sample quantitation for multiple components pesticides according to Section 7.6 of SW-846 Method 8081. Report final analyte concentration in units of micrograms per liter (µg/L). Report results that are less than 10 µg/L to 1 significant figure, and results that are greater than or equal to 10 µg/L to 2 significant figures.

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 7B: Target Compound List, Contract Required Quantitation Limits (CRQLs), and Regulatory and Spiking Levels for TCLP Chlorinated Pesticides by

SW-846 Method 8081

Analyte	**CRQL µg/L**	**Regulatory Level mg/L**	**Spiking Level µg/L**
Chlordane (Technical)	0.5	0.03	5
Endrin	0.1	0.02	1
Heptachlor	0.05	0.008	0.5
Heptachlor epoxide	0.05	0.008	0.5
Lindane (gamma-BHC)	0.05	0.4	0.5
Methoxychlor	0.5	10	5
Toxaphene	5	0.5	50

Table 8. Summary of Calibration Procedures for TCLP Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by SW-846 Method 8081

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 3 points for each analyte) (ICAL) [a, b, c]	Initially; whenever required, due to failure of CCV	RSD for CFs #20% (#30% for Surrogate compounds)	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL	Beginning of each day, after every 10 samples, and end of run	%D between CF of CCV and avg CFs from ICAL #25%	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Endrin and 4,4'-DDT Breakdown	Beginning and end of analytical sequence	#20% each analyte or #30% combined analytes	1. Investigate source of the problem and document 2. If either Endrin, 4,4'-DDT, or their breakdown products were detected, re-analyze the samples

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $5:1. If this requirement cannot be met, the laboratory must submit a method detection limit (MDL) study as part of the data package.

[b] For ICAL, prepare individual standard mixtures A and B (IND A and IND B) containing the single component pesticides specified in Table 9 of SW-846 Method 8081 at three concentration levels. For multiple response pesticides, including toxaphene and Aroclors (except 1016 and 1260), prepare separate ICAL standards at the following concentration levels: Aroclors (except 1221)at 100 ng/mL; Aroclor-1221 at 200 ng/mL; and toxaphene at 500 ng/mL. Aroclor-1016 and Aroclor-1260 may be combined into a single standard solution. Spike all calibration standards with the surrogate compounds discussed in the following Table 9 at a concentration of 20 ng/mL.

[c] Report the retention time (RT) window for each analyte. For multiple component pesticides, calculate the RT window for 5 major peaks from the initial calibration standard analysis.
Determine RT windows for both single and multiple component pesticides using the following guidelines:

Column Type	RT Window in Minutes
Packed Column	#± 2%
Mega bore or wide bore capillary column	±0.05 for tetrachloro-m-xylene through Aldrin ±0.07 for compounds which elute after Aldrin ±0.1 for decachlorobiphenyl

Table 9. Summary of Internal Quality Control Procedures for TCLP Organochlorine Pesticides and Polychlorinated

Biphenyls (PCBs) by SW-846 Method 8081

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG [a] (1 per 20 samples minimum) or one per batch of extraction fluids.	< CRQL for each compound	1. Investigate source of contamination and document 2. Re-extract and re-analyze all samples processed with a non-compliant method blank
Surrogate [b]	Every standard, sample, method blank and QC sample at 10 times CRQL	60-150% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries
Matrix Spike and Matrix Spike Duplicate (MS/MSD) [c]	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	65-135% of expected value; #30 RPD between MS and MSD	1. Address in narrative

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

[b] Spike each standard, sample, and blank with 1mL of a solution containing 0.2 µg/mL each of tetrachloro-m-xylene and decachlorobiphenyl

[c] Spike MS/MSD samples with 1mL of a solution containing the following compounds and levels:

Target compound	Concentration (µg/mL)	Target Compound	Concentration (µg/mL)
(-BHC	0.5	Heptachlor	0.5
4,4'-DDT	1.0	Aldrin	0.5
Endrin	1.0	Dieldrin	1.0

Dilute and re-analyze samples with one or more analytes at concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Second column confirmation is required for all positive results. Perform confirmation analyses on a column of a phase different from that used for quantitation. Confirmation analyses must meet all instrument calibration criteria and blank acceptance criteria specified in Table 8, above.

Table 10A. Summary of Holding Times and Preservation for TCLP Chlorinated Herbicides by SW-846 Method 1311

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation** [b]
Chlorinated Herbicides	Technical for TCLP Extraction: 14 days from collection; Contract for TCLP Extraction: 10 days from receipt at laboratory; Technical and Contract of TCLP Extract: 7 days from date of TCLP extraction to preparative extraction; Technical for Analysis: 40 days from preparative extraction; Contract for Analysis: 35 days from preparative extraction.	Cool to 4°C ±2°C

[a] Individual target compounds are listed in Table 10B.
[b] Preservatives should not be added to samples before extraction.

Data Calculations and Reporting Units:

Determine the percent solid as specified in Section 7.1 of SW-846 Method 1311 and report the result as percent solid. Extract the second sample/aliquot according to Section 7.3 of SW-846 Method 1311.

Calculate calibration factors and sample results according to Sections 7.7 and 7.8 of SW-846 Method 8151B, Chlorinated Herbicides by Gas Chromatography, (Revision 1). Report final analyte concentration in units of micrograms per liter (µg/L). Report results that are less than 10 µg/L to 1 significant figure, and results that are greater than or equal to 10 µg/L to 2 significant figures.

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 10B. Target Compound List, Contract Required Quantitation Limits (CRQLs), and Regulatory and Spiking Levels for TCLP Chlorinated Herbicides by SW-846 Method 8150B

Analyte	**CRQL µg/L**	**Regulatory Level mg/L**	**Spiking Level µg/L**
2,4-D	12	10	60-100
2,4,5-TP (Silvex)	7	10	35-100

Table 11. Summary of Calibration Procedures for TCLP Chlorinated Herbicides by

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) [a, b, c]	Initially; whenever required, due to failure of CCV	RSD for CFs #20%; or, if using a linear calibration curve, a correlation coefficient (r) of $0.99 for each compound	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL (Separate source from ICAL standards)	Beginning of each 12-hour time period, after every 10 samples and end of run	%D between calculated and nominal amount for each compound must be #±25.0%	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Retention time evaluation for CCV standards	Each analysis of CCV standards	±3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $5:1. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

[b] Report the retention time window for each analyte. Determine retention time windows as ±3 x the standard deviation (SD) of the average initial calibration retention time for each analyte.

[c] ICAL and continuing CAL standards must contain all surrogate compounds and target analytes listed in Table 10B.

Table 12. Summary of Internal Quality Control Procedures for TCLP Chlorinated Herbicides by SW-846 Method 8151

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG [a] (1 per 20 samples minimum) per analytical instrument	< CRQL for each compound	1. Investigate source of contamination and document 2. All samples processed with a method blank that is out of control must be re-extracted and re-analyzed
Surrogate Spike	Every standard, sample and method blank at 10 times CRQL	75-125% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries 2. If re-analysis does not solve the problem, re-extract and re-analyze
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum) containing all analytes chosen from Table 10B	40-160% of expected value for dinoseb and 65-135% of expected value for other target analytes; #30 RPD between MS and MSD	1. Report in Case Narrative
Laboratory Control Sample (LCS)	One LCS per batch or SDG	40-160% for dinoseb; 80-120% for other target analytes	1. Re–extract and re-analyze all samples processed with an out-of-control LCS

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

Dilute and re-analyze samples with concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Second column confirmation is required for all positive results. Confirmation must be performed on a column of a phase different from that used for quantitation. Confirmation analyses must meet all calibration criteria specified in Table 2 and blank acceptance criteria specified in Table 3 of the SW-846 Method 8151.

Table 13A. Summary of Holding Times and Preservation for TCLP Metals by SW-846 Method 1311

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation**
Metals (except mercury)	Technical: 180 days from date of collection to TCLP extraction and another 180 days from date of TCLP extraction to analysis; Contract: TCLP extraction 26 days from sample receipt at laboratory and analysis within 26 days of extraction	Cool to 4°C ±2°C After extraction and filtration, pH <2 with nitric acid
Mercury	Technical: 28 days from date of collection to TCLP extraction and another 28 days from date of TCLP extraction to analysis; Contract: TCLP extraction 26 days from sample receipt at laboratory and analysis within 26 days of extraction	Cool to 4°C ±2°C After extraction and filtration, pH <2 with nitric acid

[a] Individual target compounds are listed in Table 13B.

Data Calculations and Reporting Units:

Calculate the sample results according to the protocols of the appropriate analytical method: SW-846 Method 6010B (ICP) Section 7.6, SW-846 Methods 7470/7471 (CVAA) Sections 7.5 and 7.6, respectively, and SW-846 Method 1311 (TCLP Extraction) Section 7.2.14.

Report sample results in concentration units of milligrams per liter (mg/L).

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 13B. Target Compound List, CAS Numbers, Contract Required Detection Limits, Regulatory and Spiking Levels for TCLP Metals by SW-846 Method 6010 and SW-846 Method 7470/7471

COMPOUND	CAS No.	CRDL (mg/L)	Regulatory Level (mg/L)	Spiking Level (mg/L)
Arsenic	7440-38-2	0.50	5.0	2.5 - 5.0
Barium	7440-39-3	1.0	100	50 - 100
Cadmium	7440-43-9	0.10	1.0	0.5 - 1.0
Chromium	7440-47-3	0.50	5.0	2.5 - 5.0
Lead	7439-92-1	0.50	5.0	2.5 - 5.0
Mercury	7439-97-6	0.02 [a]	0.2	0.1 - 0.2
Selenium	7782-49-2	0.10	1.0	0.5 - 1.0
Silver	7440-22-4	0.50	5.0	2.5 - 5.0

[a] Mercury analysis is to be performed using 10 mL aliquots diluted to 100 mL. The CRDL has been adjusted to account for this 10 X dilution.

Table 14A. Summary of Calibration Procedures for TCLP Metals by SW-846 Method 6010

Calibration Element	Frequency	Acceptance Criteria	Corrective Action

Initial Calibration (minimum blank + 1 calibration standard) (ICAL)	Initially, Daily; whenever required, due to failure of CCV	Acceptable ICV, CRDL, and ICB standards	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	±10% from expected concentration	1. Terminate analysis and identify and document problem 2. Reprep and re-analyze ICV and all associated samples 3. Re-calibrate and re-analyze reprepped ICV and all associated samples
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine Source of contamination 3. Reprep ICB and CCB 4. Re-analyze all samples associated with a contaminated blank
Continuing Calibration Verification (CCV)	Before samples, after every 10 samples, and end of run	± 10% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last acceptable CCV
Contract Required Detection Limit Verification Standard (CRI)	After ICV and before sample analysis	±35% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last compliant CCV
ICP Interference Check Sample (ICS)	Run at start and finish of daily run or twice per 8 hours	± 20% from true value concentration	1. Reprep and re-analyze standard 2. Re-calibrate, verify and re-analyze all associated samples

Table 14B. Summary of Calibration Procedures for TCLP Mercury by SW-846 Method 7470/7471

Calibration Element	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Initial Calibration (minimum blank + 5 standards) (ICAL) [a]	Initially, each analytical batch; whenever required, due to failure of CCV	r $ 0.995	1. Terminate analysis 2. Re-calibrate and verify b analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	±20% from expected concentration	1. Terminate analysis and id document problem 2. Reprep and re-analyze ICV associated samples 3. Re-calibrate and re-analy ICV and all associated sa
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine source of conta 3. Reprep ICB and CCB 4. Re-analyze all samples as with a contaminated blank
Continuing Calibration Verification (CCV)	Before Samples, after every 10 samples, and end of run	±20% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back t acceptable CCV
Contract Required Detection Limit Verification Standard (CRA)	After ICV, and before sample analysis	± 35% from expected concentration	1. Reprep and re-analyze sta 2. Re-calibrate and verify

[a] The ICAL low standard must be at the CRDL.

Table 15. Summary of Internal Quality Control Procedures for TCLP Metals Analysis by SW-846 Method 6010 and SW-846 Method 7470/7471

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per SDG [a] or per batch of extraction fluid [b]	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all affected samples must be prepared again with another method blank and re-analyzed
Duplicate Sample (DUP)	One per batch or SDG [a, b]	RPD <± 20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"
Matrix Spike Sample (MS)	One per batch or SDG [a, b, c]	± 25% from expected value [d]	1. A post-digestion spike must be performed for analytes that exceed limits.
Laboratory Control Sample (LCS)[e]	One per SDG [a] or per batch of extraction fluid [b]	± 20% from expected concentration	1. Terminate analysis and identify and document the problem 2. Re-analyze all associated samples
Serial Dilution Sample (5 X Dilution) (ICP only)	One per batch or SDG [a, b]	± 10% difference from original results for analytes greater than 50 X IDL	1. Flag associated data with a "B"

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

[b] Minimum requirement is the analysis of 1 QC sample per 20 samples.

[c] Spiking solution must contain all analytes within the spiking ranges listed in Table 13B. Matrix spikes are to be added after filtration of the TCLP extract and before acidification.

[d] An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of 4.

[e] LCS spike solution must be from a different source than the calibration standards.

Dilute and re-analyze samples with concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.